



Thin Film Heat Flux Sensor Development for Ceramic Matrix Composite (CMC) Systems

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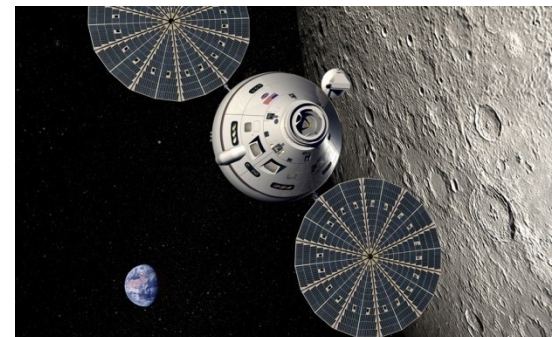
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Outline

- **Introduction**
 - NASA Projects' Needs
 - GRC Physical Sensors, Thin Films
- **High Temperature Heat Flux Sensor Foundations**
 - Designs/Applications
 - CEV TPS, Advanced Stirling Convertors, EBC-CMC
- **Sensor Material Selection**
 - Silicides, Oxides, Nanocomposites
- **Sensor Patterning**
 - EBC-CMC Surface Challenges
- **Ceramic Heat Flux Sensors on α -SiC**
 - Preliminary Results
- **Summary**



NASA's Aviation Safety Program's Aircraft Aging & Durability Project

- **Physical Issues for Life Prediction of Engine Hot Section:**

- Centrifugal Stress
- Thermal Stress
- Vibrational Stress from gas flow
- Contact Stresses from different materials (Thermal Expansions, Deformations)
- Blade Clearance (Creep)



NASA Fundamental Aeronautics Program's Supersonics Project

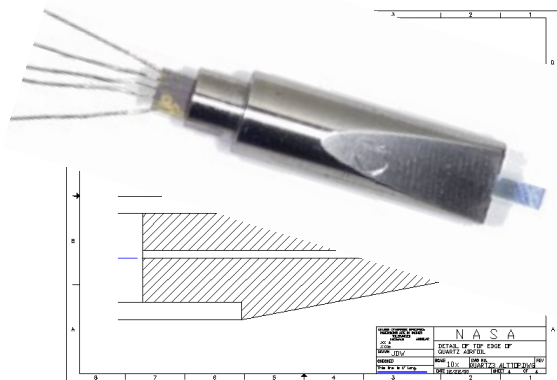


- Weight and temperature barriers for all supersonic vehicles
 - Space vehicles (launch/reentry) to military and commercial transport
 - Eliminate factors that limit efficiency and performance of supersonic vehicles
 - Application to hypersonic regimes as well
- Ceramic Matrix Composite (CMC) components
 - Allows high temperature operation at extended supersonic cruise times
 - Introduces significant weight reduction
 - Successful implementation of these ceramics requires reliable performance data and life prediction models

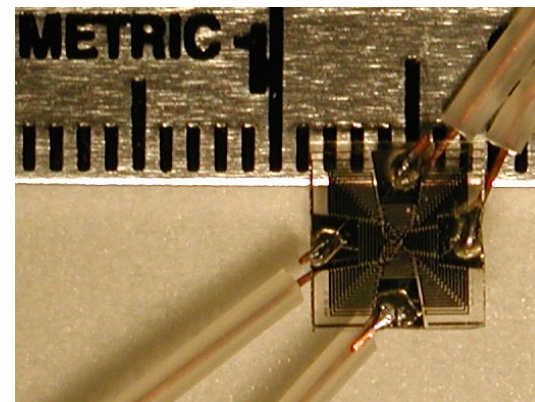


*Propulsion system
environments pose
challenges for
instrumentation*

- R&D 100 Awards in 1991, 1995, and 1998
- NASA Group Achievement Award 2003
- NASA Tech Briefs *Create the Future Design Contest* Award 2008
- Partnerships in Sensor Development:



2003 NASA Group Achievement Award
SiC High Temperature Drag Force
Transducer as part of the Integrated
Instrumentation & Testing Systems project



2008 NASA Tech Briefs Create the Future Design Contest - Machinery & Equipment



1991 R&D 100 Award
PdCr wire strain gauge applied on
Ford Motor Co. exhaust manifold



1998 R&D 100 Award
Long-lived Convolted Thermocouples
For Ceramic Temperature Measurements



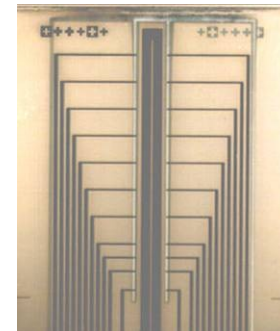
Pratt & Whitney



Thin Film Physical Sensors for High Temperature Applications

Advantages for temperature, strain, heat flux, flow & pressure measurement:

- ◆ Negligible mass & minimally intrusive (microns thick)
- ◆ Applicable to a variety of materials including ceramics
- ◆ Minimal structural disturbance (minimal machining)
- ◆ Intimate sensor to substrate contact & accurate placement
- ◆ High durability compared to exposed wire sensors
- ◆ Capable for operation to very high temperatures ($>1000^{\circ}\text{C}$)



Flow sensor made of high temperature materials

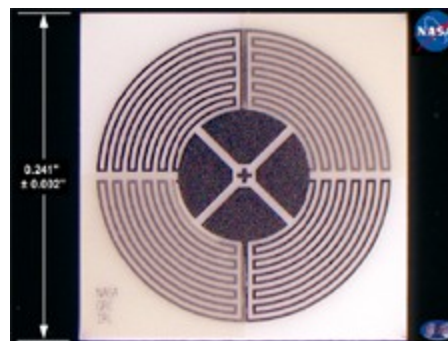
Multifunctional smart sensors being developed



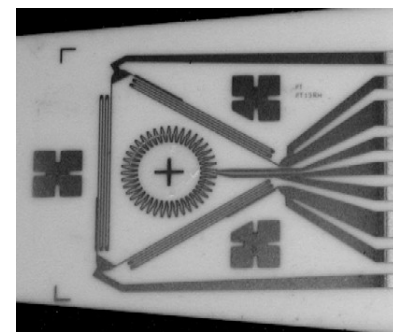
PdCr strain sensor
to $T=1000^{\circ}\text{C}$



Pt- Pt/Rh temperature
sensor to $T=1200^{\circ}\text{C}$



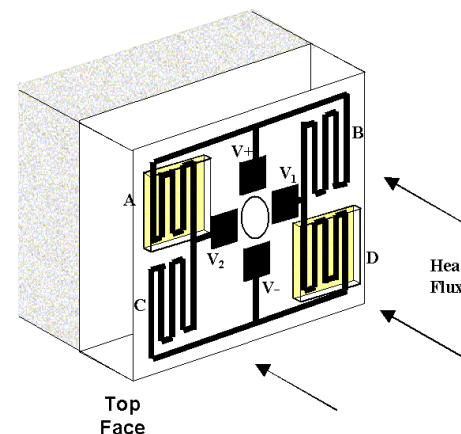
Heat Flux Sensor Array
to $T=1000^{\circ}\text{C}$



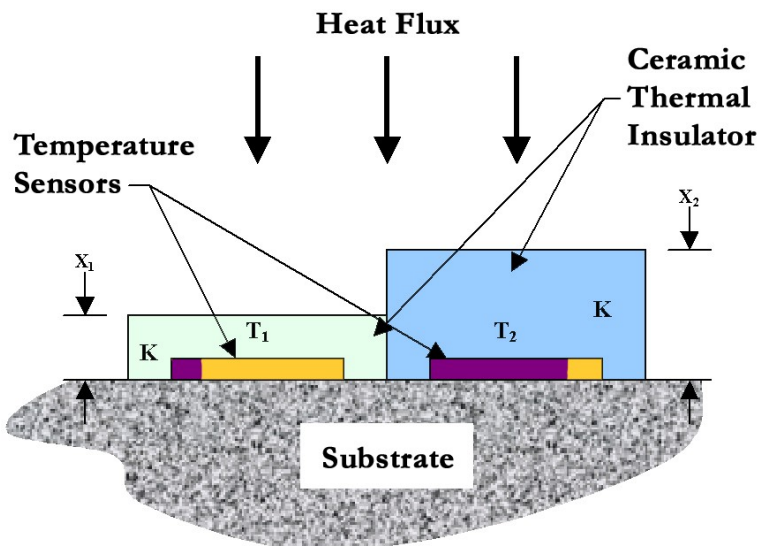
Multifunctional
Sensor Array

Basic Heat Flux Sensor

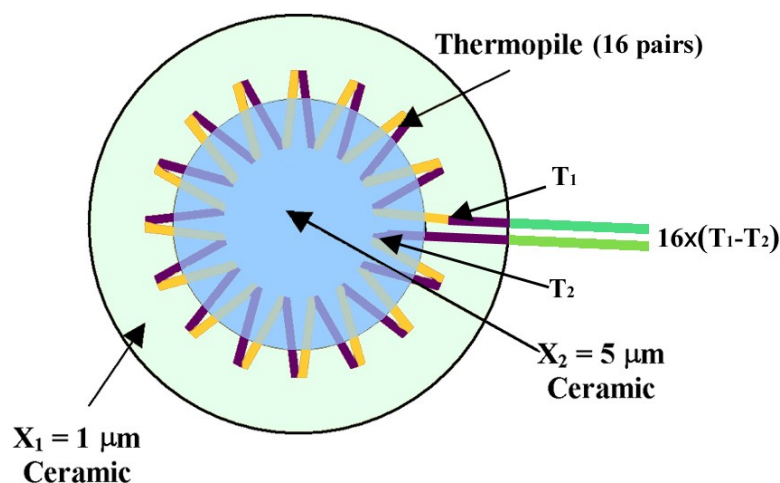
- Operates by measuring the temperature difference across a thermal resistance
- Thin film version compares temperature difference between two thicknesses of thermal insulating films
- Designs based on thin film thermopile, thermocouples and Wheatstone bridge developed at GRC
 - Example: 40-pair thermopile tested in arc-lamp up to 800°C with sensitivity of $1.2 \mu\text{V}/(\text{W}/\text{cm}^2)$ and a dynamic frequency response of 3 kHz



Wheatstone Bridge
Heat Flux Sensor Design



Heat Flux Sensor Operation

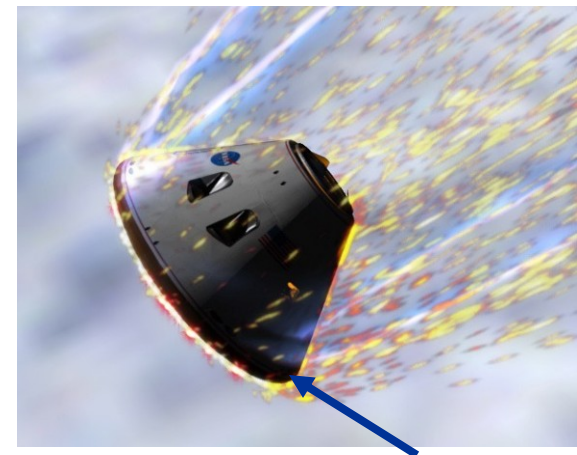


Thermopile Heat Flux Sensor Design

CEV Interface Seals Heat Flux Sensor

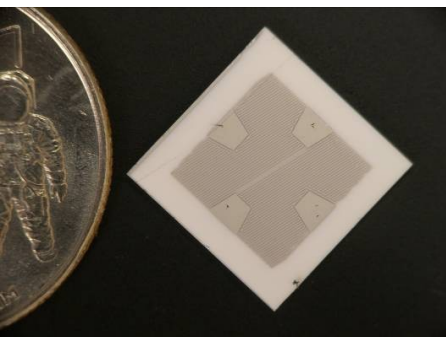
Thermal Protection System (TPS) Interface Seals

- Instrumentation for Interface Gap Heating Tests for CEV Heat Shield-to-Backshell Interface Seals
 - Design, fabrication and testing of a packaged miniature heat flux sensor
- Technology Challenge: Sensor Integration with Packaging
 - Leverage RTD Heat Flux Sensor development & Novel Thin Film Sensor effort



CEV Reentry w/ TPS Seal

Fabricated Heat Flux Sensor



0.745" Sq. Top Cover
0.020" Thick

Ceramic Cement

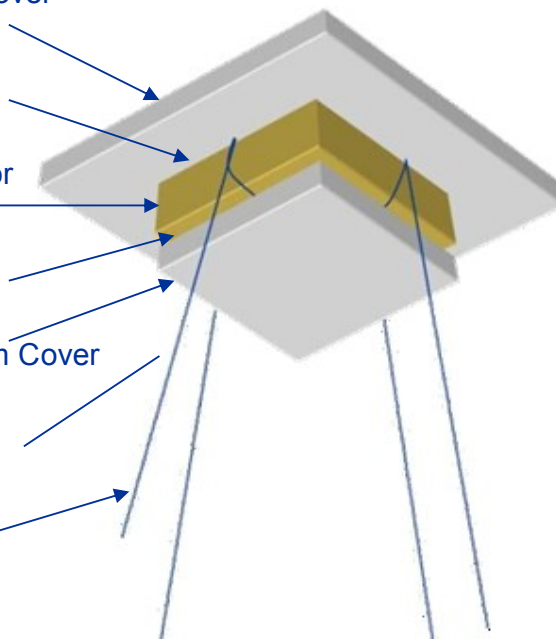
0.495" Sq. Sensor
0.040" Thick

Ceramic Cement

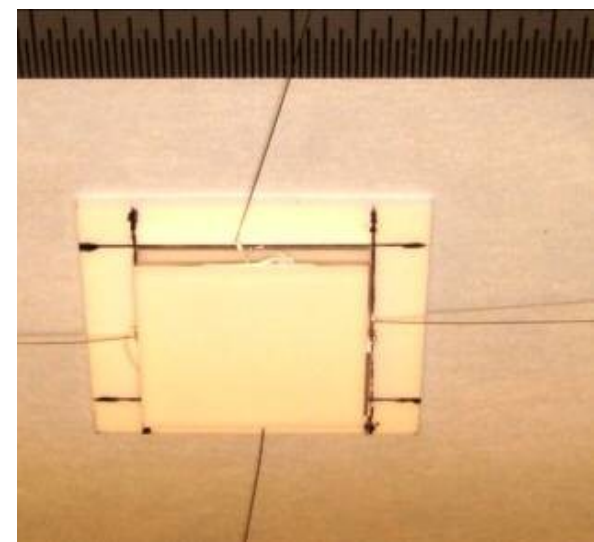
0.495" Sq. Bottom Cover
0.020" Thick

Ceramic Cement

4 x Lead Wires

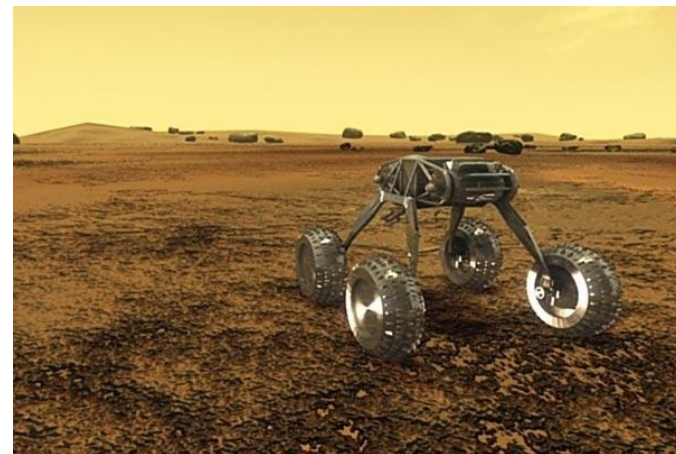


High Temperature Heat Flux Sensor (above), packaging design (right) and as packaged (far right) for CEV TPS Seal Test applications

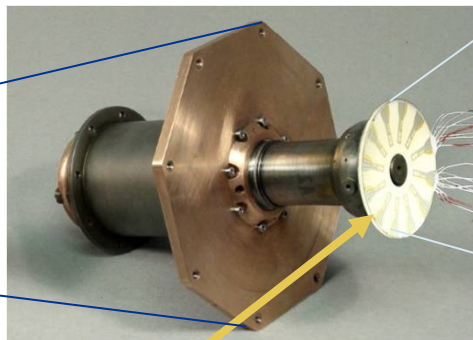
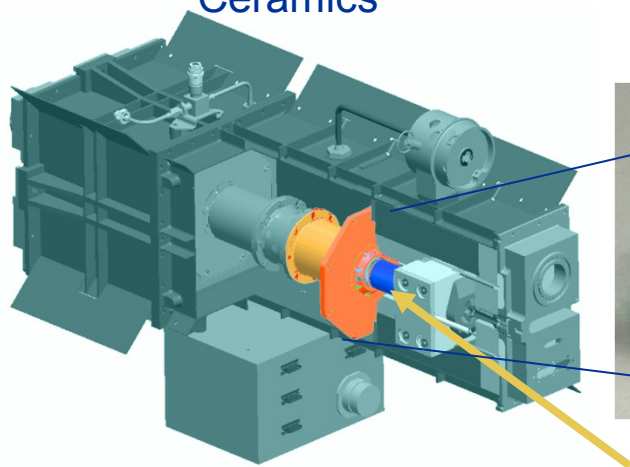


Heat Flux Sensors for Advanced Stirling Converter (ASC) Demo

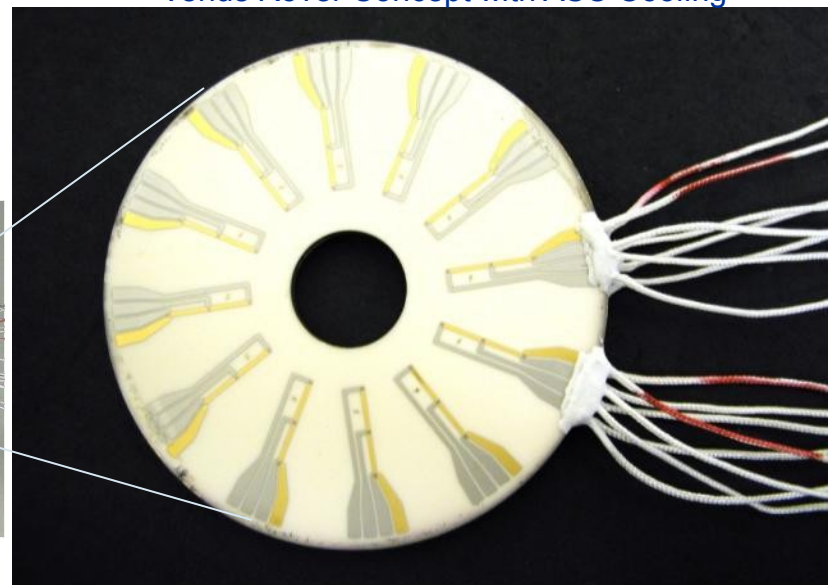
- **Development of Heat Flux Sensors for ASC-E Units** for measurement of thermal energy delivered into the converter (AIAA-2009-4581)
 - Enables a direct measurement of electrical conversion efficiency in characterizing ASC units
- Technical Challenge: Sensor Fabrication Methods Compatible with ASC Thermal and Structural Demands
 - Sensor Thermal Conductivity, Strength and Sensitivity satisfied with High Temperature Ceramics



Venus Rover Concept with ASC Cooling



ASC-E Converter

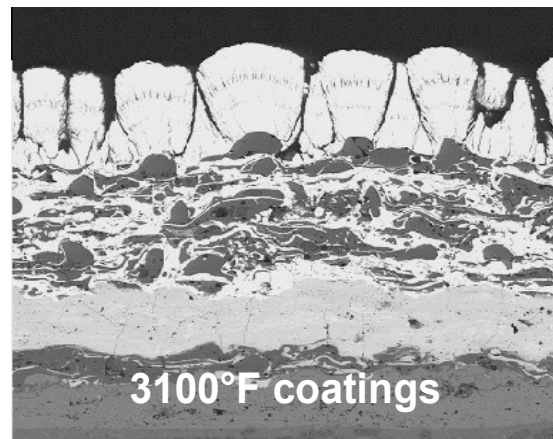


6.66 cm AuPt Heat Flux Sensor Fabricated at NASA GRC

ASRG Engineering Unit with Planned Location of Heat Flux Sensors

2700 - 3100°F SiC/SiC CMC Turbine Blade and Vane Coating System

- Multilayered coating system
 - 1700°C (3100°F) thick coating on CMC successfully completed total 100, 1 hr cycle laser heat flux test (60 min. hot, 3 min. cool)
 - 1482°C (2700°F) thin turbine blade coating under development
- In situ measurements as part of the coating system during tests will better characterize the system
 - Currently, conditions on surface measured optically
 - Conditions at interfaces currently interpolated based on bulk thermo-conductivity values
- Technical Challenge: Embedded sensors cannot use flame-sprayed attachment or coatings without modifying characteristics
 - New technology needed
 - Ceramic Thin Film Sensors?

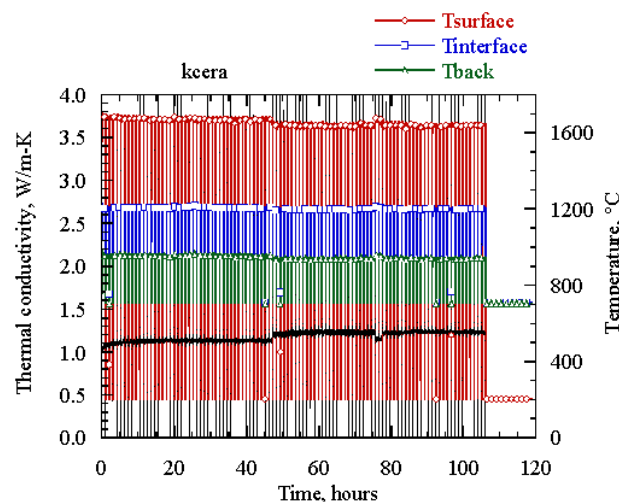


Top layer

Interlayer

EBC

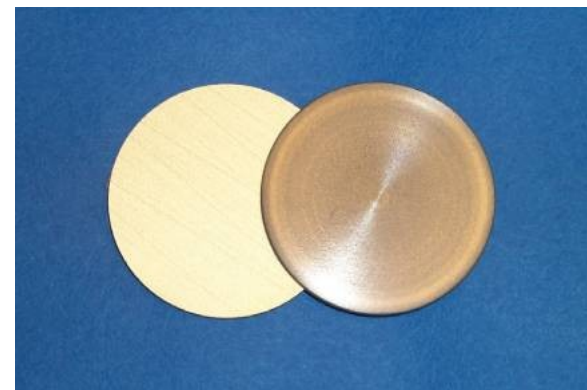
Si bond coat



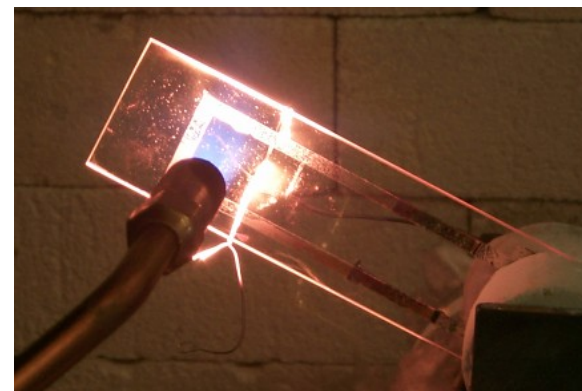
After testing

Application of Ceramics as Thin Film Sensors

- The limits of noble metal thin film sensors of 1100°C (2000°F) may not be adequate for the increasingly harsh conditions of advanced aircraft and launch technology ($>1650^{\circ}\text{C}/3000^{\circ}\text{F}$)
- NASA GRC investigating ceramics as thin film sensors for extremely high temperature applications
- Advantages of the stability and robustness of ceramics and the non-intrusiveness of thin films
- Advances have been made in ceramic thin film sensors through collaborations with the University of Rhode Island (URI)
 - Nanocomposite films are being developed by URI that show potential as thermocouples



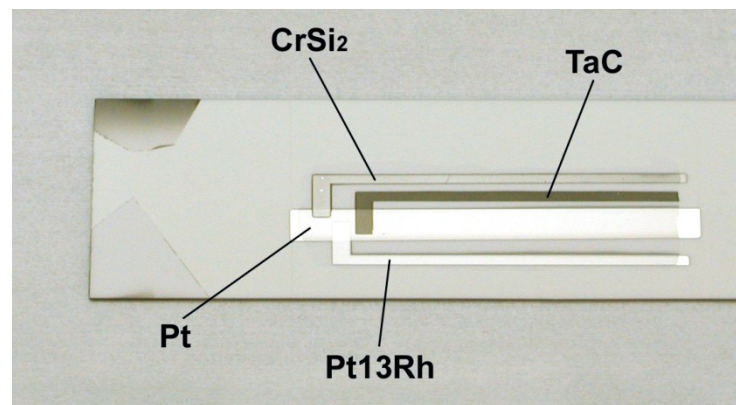
**Ceramic TC Sputtering Targets
fabricated by the NASA GRC
Ceramics Branch**



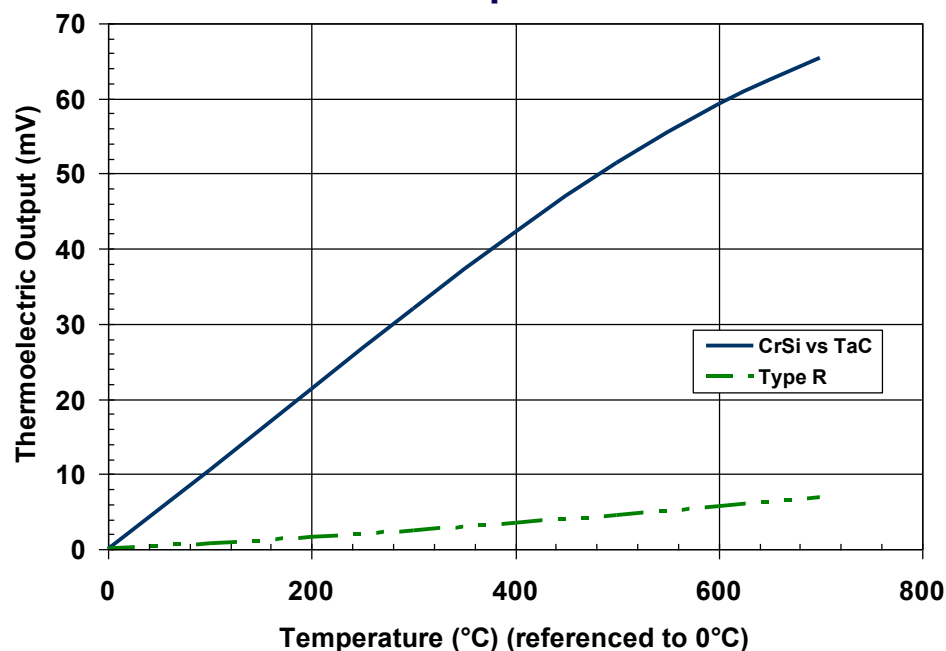
Ceramic TC fabricated at URI

Considerations for Ceramic Thermocouples

- Silicides and Carbides have highest thermoelectric output of non-metallic thermocouple (TC) elements as bulk materials
- Carbides have a very high use temperature in inert and reducing atmospheres ($>>3000^{\circ}\text{C}$)
- Silicides form a natural passivation layer in oxygen
- High Performance Silicides: CrSi, TaSi and MoSi
 - CrSi vs TaC demonstrated x12 output compared to Type R (AIAA-2004-3549)
- Next Step: Examine oxidation resistance of silicides for high temperature heat flux sensor

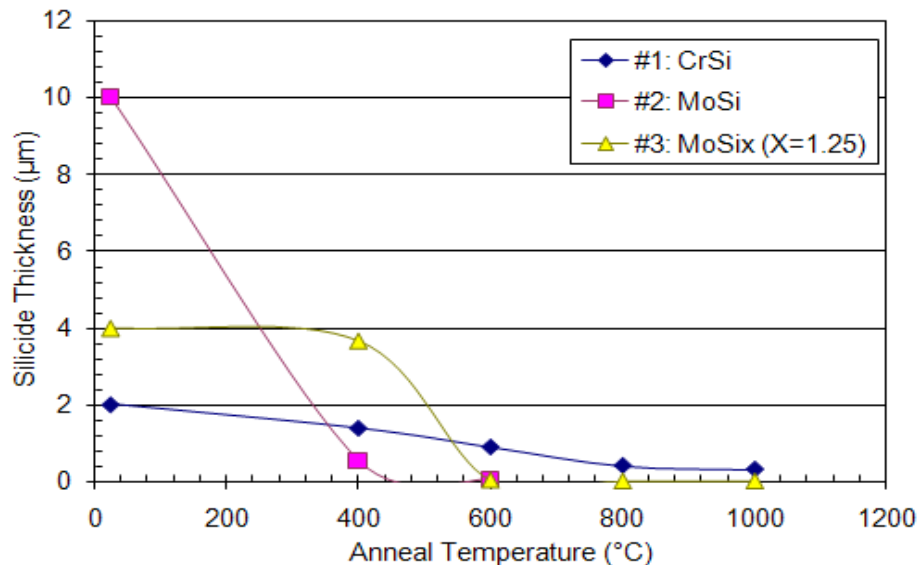


Thin Film Ceramic TC Sample and measured performance

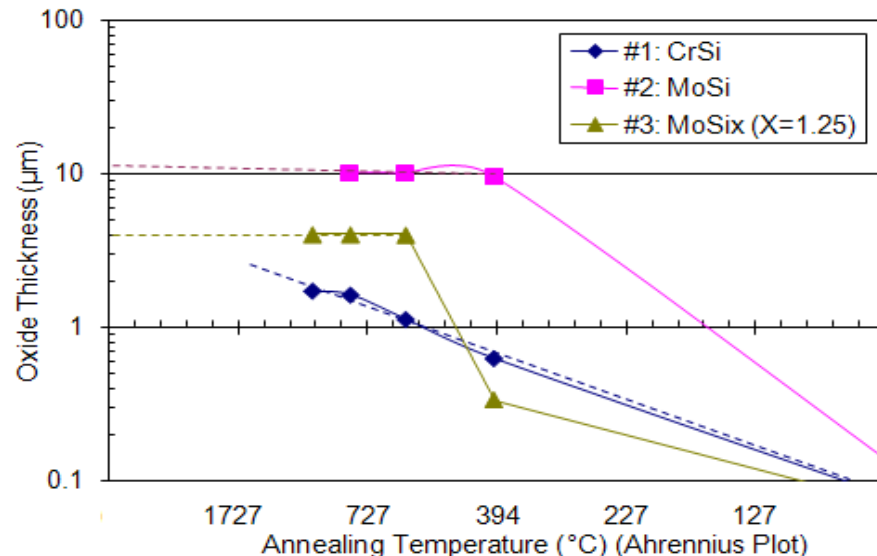


Silicide Film Thickness Tests

Silicide Thicknesses vs. Anneal Temperatures
(1 hr Annealing Time)



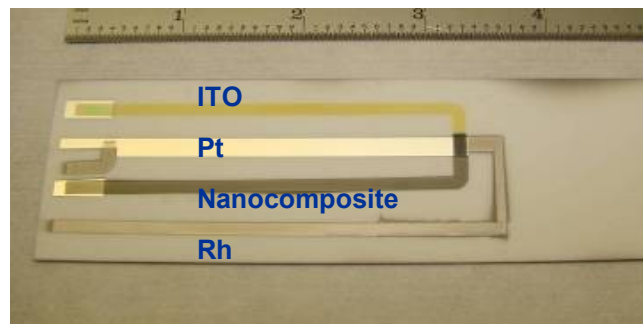
Oxide Thicknesses vs. Annealing Temperatures
(1 hr Annealing Time)



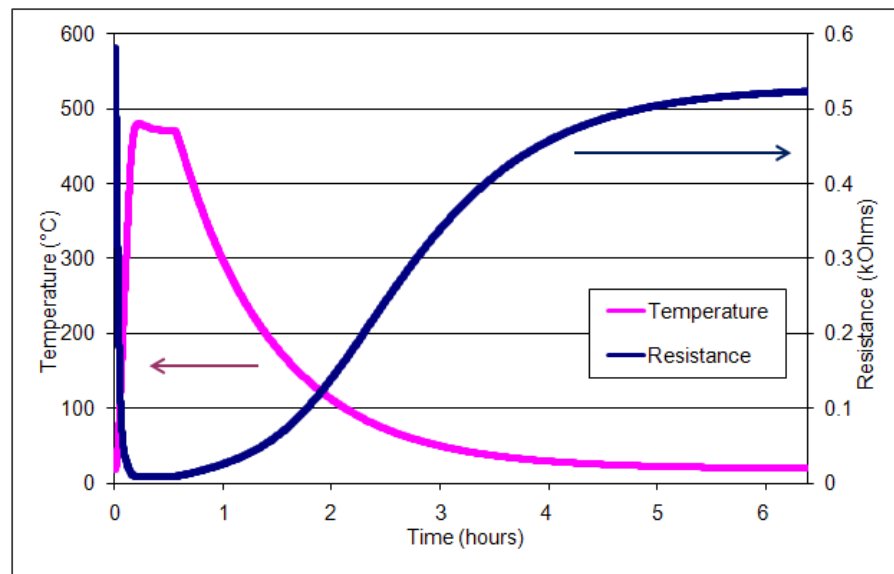
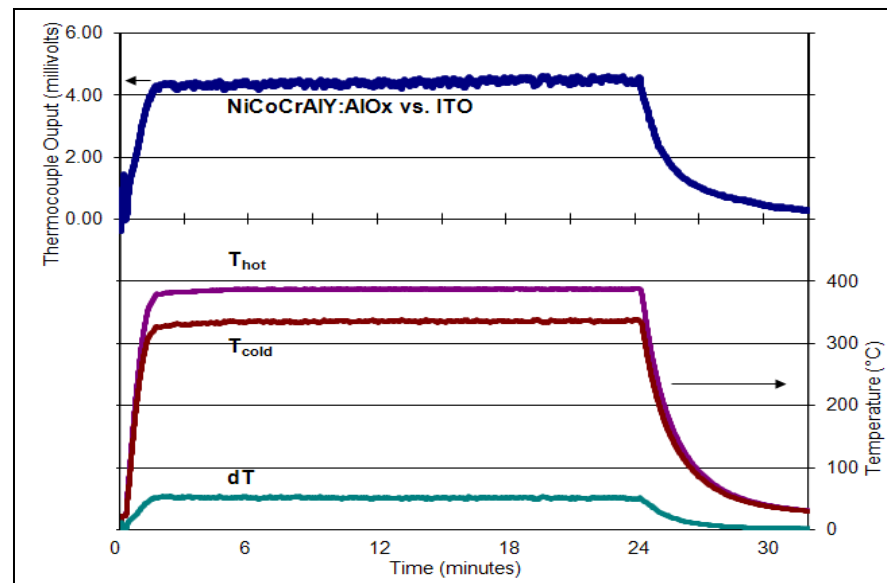
- Silicides an obvious choice on SiC CMC and/or AlSiOx EBC
- Test: CrSi and MoSi_x (x=1, 1.25) films annealed in air to evaluate oxidation performance
 - Unprotected CrSi film oxidized at a well-behaved rate
 - Unprotected MoSi_x films oxidized nearly completely over 400°C
- Some oxidation protection needed to insure film survivability
 - Embedded in EBC?
 - Conductive Oxides & Nanocomposites?

GRC Tests of Nanocomposite Sensors

- Nanocomposite of NiCoCrAlY:AlOx developed from AAD NRA w/ URI
- ITO/Nanocomposite test sample fabricated at GRC



- Tests show the 2 μm thick thermocouple stable in air with a 50 $\mu\text{V}/^\circ\text{C}$ sensitivity at 650 $^\circ\text{C}$
 - Compare Type R 12 $\mu\text{V}/^\circ\text{C}$
- The nanocomposite resistance is well-behaved with an exponential temperature response
- Primary choice for High Temperature Heat Flux Sensor on CMC
 - Either Thermopile or RTD



EBC-CMC Surface Challenges

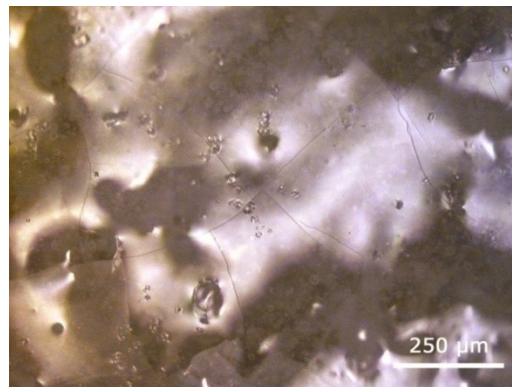
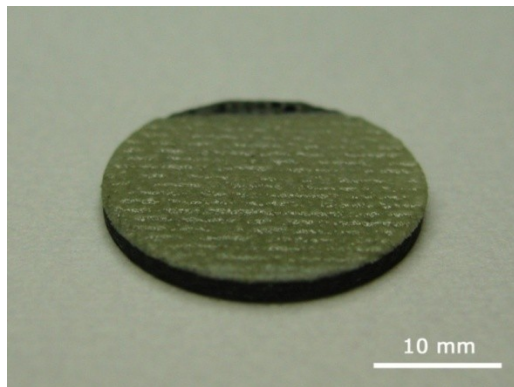
- Past Sensors on CMC without EBC
 - Wire Sensors flame-spray attachment $>1000^{\circ}\text{C}$
 - Shadow masked Thermocouples $>1000^{\circ}\text{C}$
 - Fine-line patterned sensors on AlOx flame-coated CMC $>1000^{\circ}\text{C}$
- Test articles are rough approximations of final components (part of a long-term development program)
 - CMC is naturally rough ($\sim 100\text{ }\mu\text{m}$)
 - EBC is naturally porous ($\sim 10\%$)
- Challenge is to pattern fine-lined sensors required for heat flux measurements using existing EBC-CMC materials without thick AlOx coatings
 - Smooth ($\sim 10\text{ }\mu\text{m}$) α -SiC is typically used in EBC concept tests



Wire Sensors flame-spray attachment on CMC



Fine-line sensors on AlOx-coated CMC

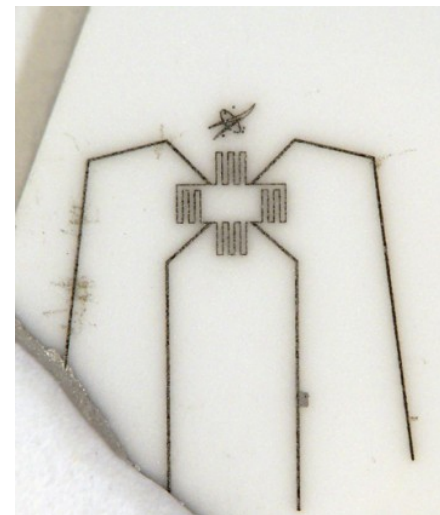


EBC-CMC:
Sample (far left)
& surface (left)



Laser Trim Patterned Heat Flux Sensor

- Fine-line 30- μm pattern possible without photolithographic techniques using Laser Ablation
- Process “direct-write” using a standard CAD file downloaded to a Nd:YAG laser machining system (1064 nm)
- Steps:
 1. Surface over-coated with aluminum
 2. Laser cut into aluminum
 3. Pt overcoat
 4. Dissolve aluminum with H_3PO_4
- Test pattern successful on smooth Alumina
- Convoluted surface of EBC-CMC made dissolving aluminum difficult



Platinum RTD Heat Flux Sensor patterned with a Nd:YAG laser on Alumina (above) and EBC-CMC (below)

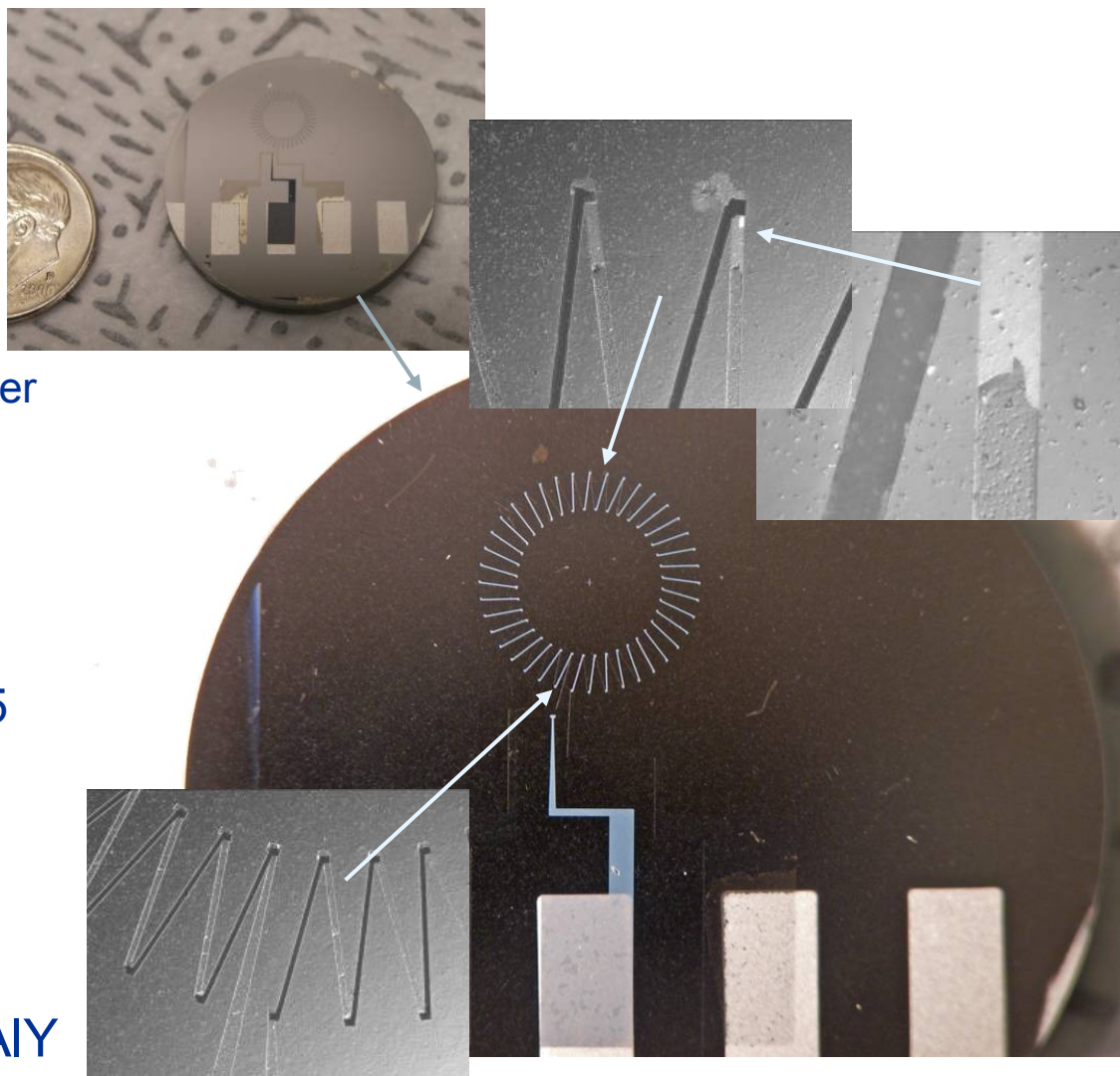


Laser Machining System



ITO-Based Heat Flux Sensor #1

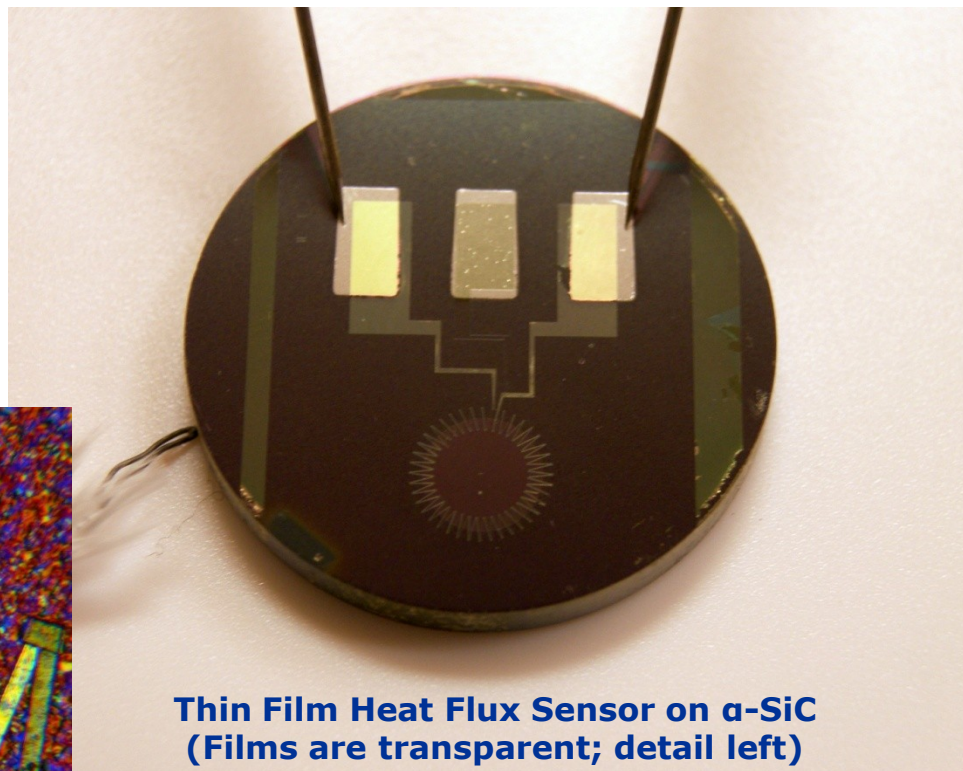
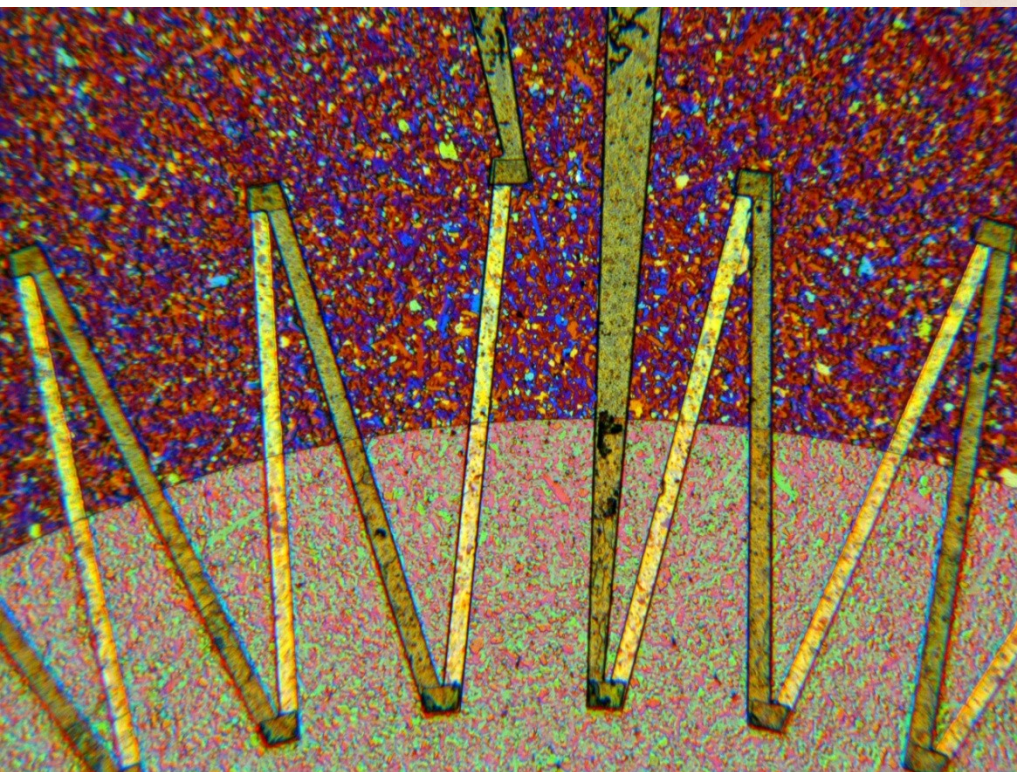
- Sensor design: Thermopile using Nanocomposite of NiCoCrAlY:Al₂O₃ vs. ITO
- Film Failures during fabrication
 - Nanocomposite films failed after 800°C in vacuum
 - ITO survived very well
- CTE mismatch may be the issue
 - NiCoCrAlY:Al₂O₃: 12 to 15 ppm/°C
 - ITO: 9 ppm/°C
 - EBC-CMC: 4.4 ppm/°C
- Next attempt: ZnO (CTE: 5 ppm/°C) instead of NiCoCrAlY



NiCoCrAlY:AlO_x-ITO Thermopile Heat Flux Sensor on α -SiC; details show film pattern after 800°C in vacuum

ITO-Based Heat Flux Sensor #2

- Fine-lined 50- μm thermopile using Al:ZnO vs. ITO on 1" disk of α -SiC
 - ITO deposited at GRC
 - Al:ZnO deposited at URI
 - Mullite used as insulation

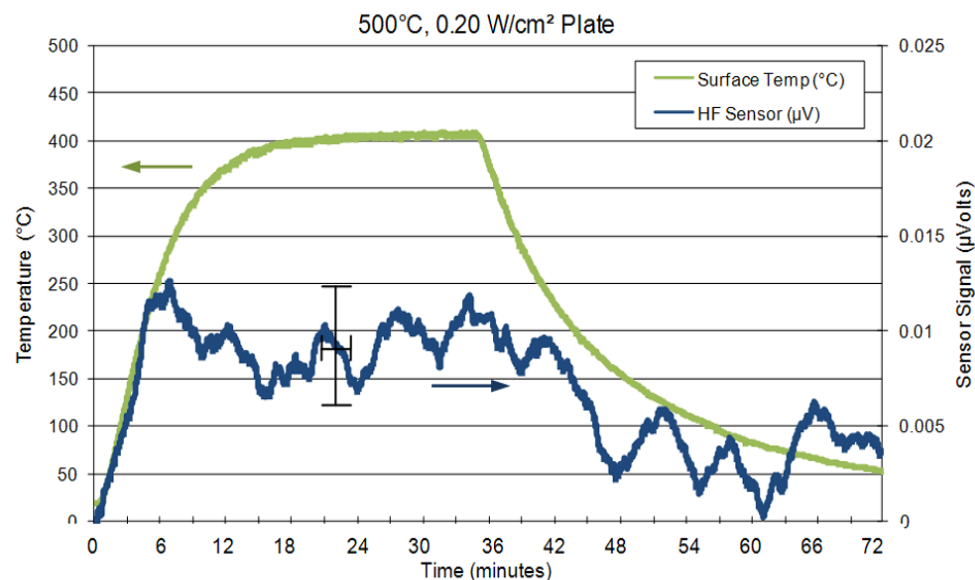
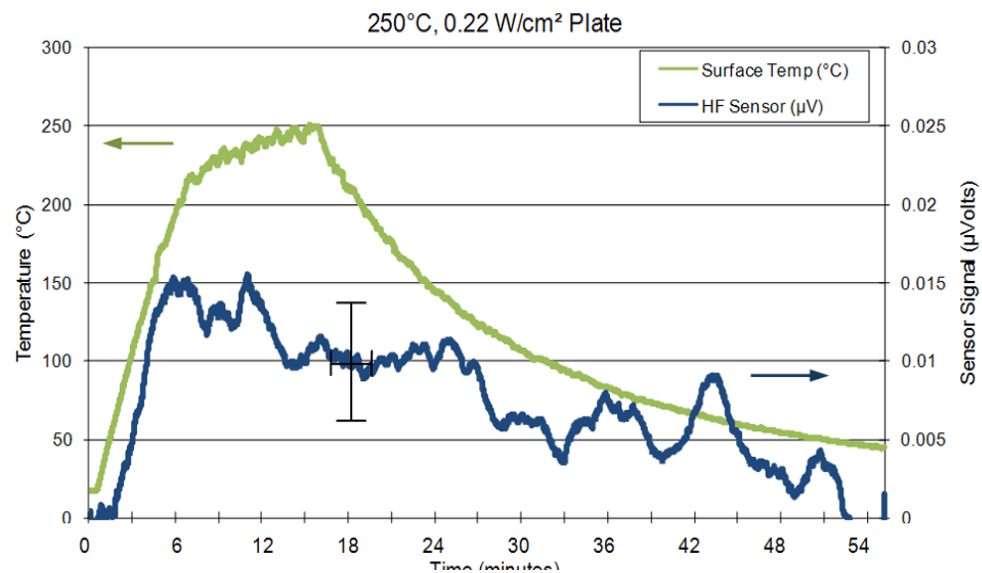


**Thin Film Heat Flux Sensor on α -SiC
(Films are transparent; detail left)**

- Sensor survived fabrication
- Response tested on two heat sources with similar heat flux but different temperatures
 - 0.22 W/cm^2 , 250°C maximum
 - 0.20 W/cm^2 , 500°C maximum

Preliminary Results

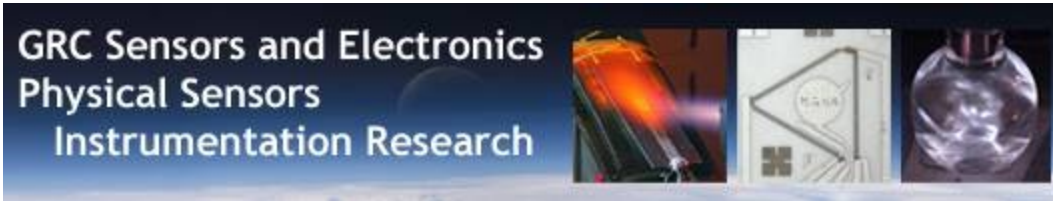
- Sensor response was very small, but noticeable
- Signals were smoothed using a moving average over 170 seconds
 - Gives minimum variation of smooth signal with original data
- The result shows a sensitivity of $0.06 \pm 0.02 \mu\text{V}/(\text{W}/\text{cm}^2)$
 - First thin film heat flux sensor pattern deposited and operated on such a surface.
 - Future sensors to increase sensor insulation to increase signal
- Future Work: Towards application on EBC-CMC
 - New, more novel materials?
 - Embedded in EBC?





Summary

- There is an increased demand for in-situ measurement of high temperature heat flux for characterizations of air and space systems
- The 1700°C EBC-CMC systems under development at GRC require such sensors to allow a more complete characterization
- The properties of silicides and conductive oxides were examined at GRC for suitability as components for a thermopile heat flux sensor
- A thermocouple of a nanocomposite film of NiCoCrAlY and alumina vs. ITO film was found to have high output over a large temperature range
- Laser-patterning was developed to be a successful method of tracing through ablation an outline of a sensor pattern in an existing film, with features $>30\text{ }\mu\text{m}$
- Heat flux sensors were fabricated with nanocomposites and conductive oxide films on the substrates of α -SiC simulating a $10\text{ }\mu\text{m}$ CMC component surface
- Best results from preliminary data was a response of $0.06 \pm 0.02\text{ }\mu\text{V}/(\text{W}/\text{cm}^2)$ was measured
- Studies with more novel sensor materials are anticipated



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<http://www.grc.nasa.gov/WWW/sensors/PhySen/>